A New Method for Measuring Elastic Moduli and the Variation with Temperature of the Principal Young's Modulus of Rocksalt Between 78°K and 273°K

LEWIS BALAMUTH, *Columbia University*

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A new method is described for measuring the elastic moduli of a homogeneous, anisotropic substance at an arbitrarily chosen temperature between 78°K and 300°K. The method is characterized by high precision and by the fact that the specimen upon which the measurement is made is small and easily prepared. Data are given which show the variation with temperature of the principal Young's modulus of rocksalt between 78°K and 273°K.

A. Theory of the Method

The theory offers a description of the behavior of an electromechanical system constructed as follows: A slender rectangular cylinder of piezoelectric quartz is so cut from the crystal that an electric axis lies perpendicular to one pair of opposite sides, and the optic axis to another. The cross-sectional dimensions of the cylinder are approximately 3 mm, and its length varies from 1 to 3.5 cm. The sides normal to the electric axis are coated with gold leaf. A cylinder of specimen material 2 or 3 cm long and of nearly identical cross section is cemented to one end of the quartz rod with suitable adhesive, and the composite body is suspended by fine threads. A harmonically varying potential difference of constant amplitude is established between the gold leaf electrodes, and means are provided for measuring the amplitude of the resulting current. In consequence of the harmonically varying piezoelectric stress in the quartz, which accompanies the electric field between the electrodes, the entire suspended body assumes a state of forced vibration, the frequency of which is the same as that of the applied potential difference. It is the object of the present theory to relate the electrical reactance between the gold leaf electrodes to the Young's modulus of the specimen material for the direction of the cylinder axis.

In the first instance we shall develop a theory which offers an approximate description of the behavior of an electromechanical system analogous to the above, but simplified in the following particulars: (1) The cross section is circular; (2) both materials are isotropic; and (3) the piezoelectric stress in the quartz is such as would be produced by equal normal surface tractions, uniform over its ends, and given by the real part of P, where

$$P=P_0e^{ikt}, \tag{1}$$

P_0 is a constant amplitude, and k is 2π times the frequency of the applied potential difference. In the following analysis quantities characteristic of the state of affairs in the quartz are designated by the subscript 2, and of that in the specimen by the subscript 1.

The equations of motion at points within the specimen and quartz rods are, respectively,

$$\nabla\cdot\Phi_i=\rho_i\partial^2\mathbf{s}_i/\partial t^2, \quad (i=1,\ 2), \tag{2}$$

where ρ_i is the density, $\mathbf{s}_i$ the vector displacement and Φ_i the stress dyadic. A complete description of the motion of the oscillator is given by two vector point functions, $\mathbf{s}_i$, which satisfy, respectively:

(I) Eqs. (2) at all points in the oscillator;

(II) the equations

$$\Phi_i\cdot\mathbf{n}=0, \quad (i=1,\ 2), \tag{3}$$

at all points on the lateral boundary of the oscillator, where $\mathbf{n}$ is an outward drawn normal;

(III) the equations

$$\Phi_2\cdot\mathbf{n}+P=0, \text{ and } \Phi_1\cdot\mathbf{n}=0, \tag{4}$$

at all points on the end surfaces of the oscillator;

(IV) the equations

$$\Phi_2\cdot\mathbf{n}+P=\Phi_1\cdot\mathbf{n}, \tag{5}$$

and

$$\mathbf{s}_1=f(\mathbf{s}_2), \tag{6}$$

at all points on the surfaces joined by the cement, where $f(\mathbf{s}_2)$ is a function of $\mathbf{s}_2$ whose form is left undetermined for the present.

As a matter of fact, functions which meet these four conditions have not been discovered.[1] We resolve the difficulty in the usual way by replacing the boundary conditions expressed by Eqs. (4), (5) and (6) with others implied by the formulae

$$\int_{A_2}(\Phi_2\cdot\mathbf{n}+P)dA=0, \tag{7}$$

and

$$\int_{A_1}(\Phi_1\cdot\mathbf{n})dA=0, \tag{8}$$

on the ends, and at the interface

$$\int_{A_2}(\Phi_2\cdot\mathbf{n}+P)dA=\int_{A_1}(\Phi_1\cdot\mathbf{n})dA, \tag{9}$$

and

$$\int_{A_1}\mathbf{s}_1 dA=\int_{A_2}f(\mathbf{s}_2)dA, \tag{10}$$

where A_i is the cross-sectional area. The significance of these substitutions is: (1) the tangential stresses at the ends, together with these and the tangential displacements at the interface, are eliminated, since by symmetry they integrate to zero; (2) the condition of continuity of total force replaces that of continuity of stress at the interface; and (3), the condition of continuity of average displacement replaces that of actual displacement at the interface. The functions $\mathbf{s}_i$ which will be discussed presently meet these modified conditions, and experiment reveals that they offer an adequate description of the motion of the oscillator.

The early part of the analysis leading to the discovery of the functions $\mathbf{s}_i$ is similar to that used by Chree[2] to obtain a description of the free vibration of an isotropic right cylinder. The method of Chree yields expressions for $\mathbf{s}_i$, which satisfy conditions (I) and (II) and Eqs. (7), (8), (9) and (10) as far as the tangential components of stress and displacement are concerned. These expressions contain four undetermined constants which are evaluated with the aid of the four equations, obtained from (7), (8), (9) and (10), relating the normal stress and displacement components over the ends and interface. The form of the function $f(\mathbf{s}_2)$ is assumed to be such that at the interface

$$\int_{A_1}\mathbf{s}_1\cdot\mathbf{n}dA=h\int_{A_2}\mathbf{s}_2\cdot\mathbf{n}dA, \tag{11}$$

where h is a constant whose value may be expected to differ only slightly from unity. The resulting expressions offer a complete description of the motion in terms of P, h, and the dimensions and physical constants of the quartz and specimen rods. The expressions are rendered usable by retaining therein terms of no higher order than the second, in the quantity a/l_i where a is the radius of either cylinder and l_i its length.

Van Dyke[3] has shown that, in consequence of the piezoelectric charge distribution accompanying the vibrational strain in the quartz, the electrical reactance between electrodes of the sort here described contains a part which depends upon the space average value of this strain. The latter quantity, which we shall denote by $\bar{s}$, is readily calculated from the foregoing solution, and is given by the expression

$$\bar{s}=\{V_0F(y_1,y_2)/\Delta\}e^{jkt}, \tag{12}$$

where V_0 = amplitude of applied voltage,

$$\begin{aligned}
y_i&=\pi(f/f_i)[1+(\tfrac{1}{2})\pi^2\sigma_i^2(\theta_i/A_il_i^2)(1-f^2/f_i^2)],\\
f&=k/2\pi,\qquad \sigma_i=\text{Poisson's ratio},\\
\theta_i&=\text{moment of inertia of the cross section about the cylinder axis},\\
l_i&=\text{length of a cylinder},\\
f_i&=n(G_i^{\frac{1}{2}}/2\rho_i^{\frac{1}{2}}l_i)(1-\pi^2n^2\sigma_i^2\theta_i/4l_i^2A_i),\\
n&=\text{an integer},\\
G_i&=\text{Young's modulus for the direction of the cylinder axis},\\
\Delta&=(M_2/hM_1y_2)\tan y_2+(1/y_1)\tan y_1,\\
M_i&=\text{mass of a cylinder},
\end{aligned} \tag{13}$$

and $F(y_1, y_2)$ is a function of the frequency, f, which has no singular points. Solutions for f of the equation

$$\Delta=0 \tag{14}$$

[1] See, for example, *The Theory of Sound* by Lord Rayleigh Vol. I, 2nd Ed., p. 113.

[2] C. Chree, Quart. J. Math. **21**, 287 (1886).

[3] Van Dyke, Proc. I. R. E. **16**, 742 (1928).

determine a set of frequencies at which the amplitude of $\bar{s}$ is very large. We shall call these frequencies "resonance frequencies" and denote them by the symbol f_0. Over a considerable frequency range in the neighborhood of f_0 the function $F(y_1, y_2)$ remains practically a constant. Restricting f to such a range, $\bar{s}$ may be written in the form

$$\bar{s}=\{V_0/B\Delta\}e^{ikt}, \tag{15}$$

in which B is a constant.

The foregoing analysis suggests the following experimental method for measuring G_1: In virtue of the before-mentioned relation between $\bar{s}$ and the electrical reactance between the electrodes, it is clear that the quantity f_0 can be measured by observing the variation of the reactance with frequency in the neighborhood of f_0. Furthermore an inspection of Δ reveals that $f_0=f_2$ when the specimen is removed from the quartz, i.e., when $\rho_1=0$. If the quantity f_2, characteristic of the quartz, be measured in this manner it follows that the only unknown quantities that appear in Eq. (14) are h and f_1. It is found that a correct description of the behavior of oscillators of the sort here dealt with is obtained by setting $h=1$, so that Eq. (14) yields directly the value of f_1. With this value of f_1, Eq. (13) may be solved for G_1 when σ_1 is known. The term in Eq. (13) which contains σ_1 is very small compared with unity, so that an accurate value of G_1 may be obtained with an approximate value of σ_1.

B. The Electrical Circuit

The electrodes on the quartz are connected to the terminals of a variable condenser in a simple resonant circuit, as shown in Fig. 2. This circuit is coupled so loosely to the output of a vacuum tube oscillator that the voltage induced therein is independent of the impedance.

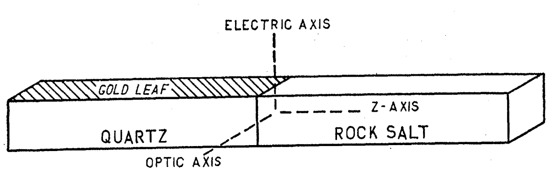

FIG. 1. The composite oscilllator.

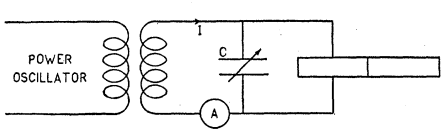

FIG. 2. The Cady circuit.

The amplitude of the current, I, which flows in the resonant circuit is measured with a vacuum thermocouple A. The applied frequency is varied in the neighborhood of the frequency f_0; and for each value thereof the capacity, C, is so adjusted that the amplitude of the current is a maximum. Cady[4] has shown that under these circumstances the least of these maximum values of I is obtained when the applied frequency is a solution of Eq. (14). This critical frequency setting is easily made, and the frequency itself, which is f_0, is then measured to five parts in a hundred thousand by beating the output of the vacuum tube oscillator against that of a piezoelectric clock in the manner described by Quimby.[5]

C. The Oscillator

At room temperature the quartz and specimen rods are cemented together under pressure, in a vacuum, with an adhesive composed of para rubber dissolved in vaseline. The oscillator is suspended vertically between rigid glass rods by means of fine threads, attached with shellac to opposite sides of the quartz near a displacement node of vibration. Thin strips of gold leaf are affixed to the electrodes with saliva, and these lead to stout contacts on the glass rods. The assembly is shown in Fig. 3. The base of the mounting is a ground glass plug which closes the bottom of an evacuated glass cylinder.

D. The Temperature Control

The observations here to be reported were made at temperatures between 78°K and 273°K. The oscillator, enclosed in the evacuated glass cylinder, together with a Leeds and Northrup platinum resistance thermometer were placed in a well stirred liquid bath held in a Dewar flask. The liquid used at temperatures above 213°K was methanol. Between 140°K and 213°K the liquid was composed of 18.1 percent chloroform, 8 percent ethyl chloride, 41.3 percent ethyl bromide, 12.7 percent trans-dichlorethylene and

[4] W. G. Cady, Proc. I. R. E. **10**, 83 (1922).
[5] S. L. Quimby, Phys. Rev. **39**, 345 (1932).

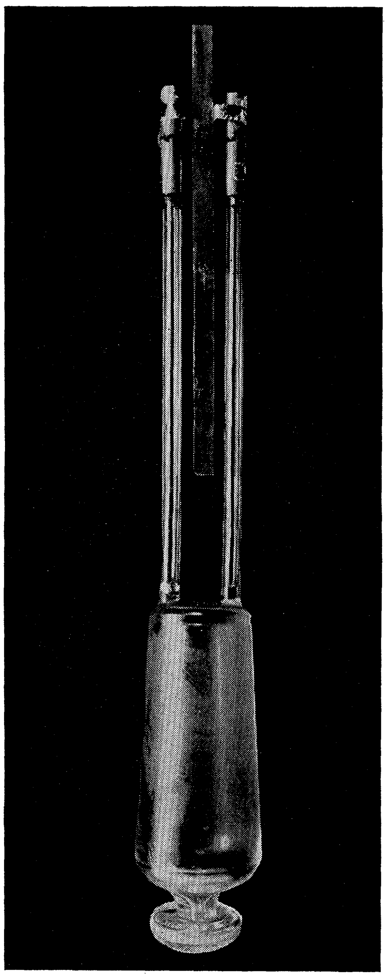

FIG. 3. A mounted oscillator.

19.9 percent trichlorethylene.[6] Percentages were reckoned by weight. Below 140°K three sets of observations were taken with the oscillator and thermometer immersed in liquid oxygen, liquid air and liquid nitrogen, respectively. The temperature of the methanol bath was stabilized within one-tenth of a degree at arbitrarily chosen values by the addition of suitable small amounts of cold liquid; and that of the other by varying the rate of flow of liquid air circulated through a copper spiral tube wound close to the inner wall of the Dewar flask. The resistance change of the thermometer was measured on a Leeds and Northrup-Mueller bridge.

[6] C. W. Kanolt. Bur. Stand. Sci. Paper No. 520 (1926).

E. RESULTS

The specimen material used in this research was the finest grade of optical rocksalt obtainable. It was secured through Ward's Natural Science Establishment, Inc., who write that it was mined near the town of Artemovsk in the Ukraine. Two groups of four specimen rods each were cut from an eleven ounce crystal. The direction cosines, α, β, γ, of the cylinder axes of these two sets of specimens were, respectively, 0, 0, 1, and 0, 0.7021, 0.7121.

The data in addition to f_1 necessary to evaluate $G_1(\alpha, \beta, \gamma)$ in accordance with Eq. (13) are the length, l_1, at a known temperature, and the density, ρ_1, and, approximately, Poisson's ratio, $\sigma_1(\alpha, \beta, \gamma)$, as functions of the temperature. l_1 was measured at room temperature with a comparator. ρ_1 is given as a function of the temperature by the formula[7]

$$\rho_1 = 2.1680(1 - 11.2\times 10^{-5}t - 0.5\times 10^{-7}t^2),$$

where t is the temperature in °C, and ρ_1 is in grams per cm^3. To calculate $\sigma_1(\alpha, \beta, \gamma)$ the three principal elastic moduli, s_{11}, s_{12} and s_{44}, must be evaluated.[8] The latter quantities are related to $G_1(\alpha, \beta, \gamma)$ by the formula[9]

$$1/G_1(\alpha, \beta, \gamma) \equiv s_{11}' = s_{11} - 2(s_{11} - s_{12} - \tfrac{1}{2}s_{44})(\beta^2\gamma^2 + \gamma^2\alpha^2 + \alpha^2\beta^2). \quad (16)$$

Values of $G_1(\alpha, \beta, \gamma)$ calculated without the term in $\sigma_1(\alpha, \beta, \gamma)$ yield approximate values for s_{11} and the combination $(s_{11} - s_{12} - \frac{1}{2}s_{44})$. A sufficiently accurate estimate of s_{44} was supplied the writer by Mr. F. C. Rose who is at present at work on the precise determination of this quantity. The values of $\sigma_1(\alpha, \beta, \gamma)$ used in the final calculation of $G_1(\alpha, \beta, \gamma)$ are given in Table I.

TABLE I. *Values of Poisson's ratio.*

T°K	$\sigma_1(0, 0, 1)$	$\sigma_1(0, 1/\sqrt{2}, 1/\sqrt{2})$
273.1	−0.25	−0.37
90.0	−0.26	−0.43

[7] Fr. A. Henglein, Zeits. f. physik. Chemie **115**, 97 (1925).

[8] W. Voigt, *Lehrbuch der Kristallphysik*, p. 631.

[9] W. Voigt, reference 8, p. 739.

TABLE II. *Average values of the elastic moduli.*

T °K	$s_{11}\times10^{13}$ cm²/dynes	a.d. of s_{11}	$s_{11}'\times10^{13}$ cm²/dynes	a.d. of s_{11}'
273.1	22.14	0.087	28.41	0.18
270	22.08	0.087	28.33	0.16
260	21.86	0.085	28.22	0.19
250	21.65	0.076	28.10	0.17
240	21.43	0.066	27.99	0.16
230	21.21	0.070	27.86	0.17
220	21.02	0.061	27.74	0.17
210	20.82	0.063	27.62	0.17
200	20.63	0.067	27.51	0.17
190	20.43	0.068	27.40	0.16
180	20.24	0.061	27.30	0.17
170	20.06	0.058	27.21	0.17
160	19.89	0.055	27.11	0.17
150	19.72	0.052	27.02	0.17
140	19.55	0.051	26.94	0.17
90	18.77	0.048	26.58	0.16
80	18.64	0.043	26.51	0.16
	$\alpha=\beta=0,\ \gamma=1$		$\alpha=0,\ \beta=0.7021,\ \gamma=0.7121$	

Table II gives the average values of the elastic moduli associated with the rod axis for the two groups of four specimens each.

F. Precision

The platinum resistance thermometer used in the experiments was calibrated by the Bureau of Standards, and was checked against another likewise so calibrated. The only source of difference between the temperature of the oscillator and that of the bath surrounding it arises from a vertical temperature gradient along the cylinder in which the oscillator is mounted. It was shown without difficulty that this vanishes quite close to the surface of the bath. Repeated observations on the same oscillator over the entire temperature range yielded values of f_0 in agreement to 0.01 percent. Variations in f_0 accompanying the reconstruction of the assembly shown in Fig. 3 from the same elements were never greater than 0.05 percent.

The accuracy of a measurement of l_1 was 0.02 percent, and that of the orientation of a cylinder axis was $\pm 8'$ of arc. The overall uncertainty in the absolute values of s_{11} and s_{11}' is thus considerably less than the variations in these quantities observed among different specimen rods *cut from the same crystal.* The latter are indicated by the figures given in the a.d. columns of Table II.

G. Discussion of Results

The only other attempt to determine the temperature dependence of s_{11} for rocksalt, in the range reported in this paper, was made by Steinebach.[10] Using a static method he measured the ratio of s_{11} at room temperature to its value at the temperature of liquid air. These temperatures in Steinebach's work were 291°K and 88°K. His value for the ratio is 1.195. The corresponding ratio formed from Table II is 1.199.

Voigt[9] measured s_{11} statically at room temperature and hence obtained the isothermal modulus, while the values of s_{11} here reported are adiabatic. According to a theory by Voigt,[11] the adiabatic modulus should be about 1 percent lower than the isothermal for rocksalt. Table II shows a value of s_{11} about 5 percent lower than Voigt's value when compared at room temperature. In this connection it should be noted that Voigt's rocksalt came from Stassfurt. As a matter of fact, measurements made by the present method upon various specimens of rocksalt of unknown origins yielded values of s_{11} which bracketed that of Voigt.

H. Discussion of Method

The electromechanical system whose behavior is described by the formulae of the theory differs from that upon which the experiments were performed in the following particulars: (1) neither material is isotropic; (2) the actual piezoelectric stress in the quartz is not of the assumed sort; (3) the cross section is not circular; (4) both materials possess internal friction; (5) the system is constrained by its support; (6) the layer of adhesive has inertia.

Additional sources of error in the description are: (1) the substitution of integral for point boundary conditions over the end areas and interface; (2) the assumption of a linear displacement relationship over the interface.

None of these circumstances vitiates the method. Various arguments in support of this assertion might be adduced from the analysis of the free vibration of crystalline rods developed by

[10] T. Steinebach, Zeits. f. Physik **33**, 674 (1925).
[11] W. Voigt, reference 8, p. 789.

Chree[12] and von Laue.[13] It is preferable, however, to justify the method experimentally as far as possible.

A simple procedure demonstrates that no factor concerned with the quartz is a source of error in these experiments. Values of f_1 were obtained with a succession of oscillators constructed by cementing the *same* specimen rod to various quartz rods of widely differing lengths. Such experiments invariably reveal that this quantity, as calculated with the formulae of the theory, is in fact characteristic of the specimen rod and the adhesive.

Cognizance is taken of the adhesive in an alternative theory in which this layer is regarded as a part of a tripartite oscillator. The formulae of this theory show that the effect of the adhesive is small for all values of f_0 except those that lie in the neighborhood of a value which causes a displacement node of vibration to appear at the interface. If the length of the specimen bar is so adjusted that $f_0=f_1=f_2$ within ± 10 percent of their values, then the effect of the adhesive is negligible. (Such adjustment is always made.) These conclusions are verified in the first instance by comparing values of f_1 obtained when the same units are joined with various adhesives having very different elastic and inertial properties. Further verification is supplied by the observations reported in the next paragraph.

It is desired next to show that the quantity G_1 as calculated with Eq. (13) is, to the order $a^2/4l_1^2$, characteristic of the substance of which the specimen rod is composed and of the orientation of the cylinder axis. This is accomplished by comparing values of G_1 obtained with a succession of oscillators constructed by cementing specimen rods of widely varying lengths to the same quartz rod. The different specimen rods are obtained by continued amputation of a single rod, initially long. The procedure leaves the substance and orientation unchanged, but alters f_1 and the value of the second term on the right-hand side of Eq. (13).

Lastly, it must be shown that the quantity G_1 calculated with Eq. (13) is, in fact, Young's modulus. If the cross-sectional dimensions of the specimen cylinder were indefinitely small compared with its length, this result would follow from Hooke's law and Newton's laws of motion. It is assumed that the experimental value of G_1, which is invariant to an alteration in the value of $a/2l_1$ between 0.05 and 0.03, is the value which would be measured if $a/2l_1$ were zero.

Table III contains typical data obtained from a set of experiments of the sort described above. Two specimen rods for each orientation were obtained, one from the other, by amputation. Their lengths were, respectively, 2.8216 cm and 2.0342 cm for the 100 orientation, and 3.4752 cm and 2.3275 cm for the 110 orientation. Eight different oscillators were constructed by cementing these specimens, in succession, to two quartz rods of lengths 3.21936 cm and 2.4987 cm. (All these lengths are at 273.1°K.) The values of f_2 for these quartz rods are 86,691 and 111,517 at 273.1°K. The elastic moduli were measured at 273.1°K and 85°K. $f_0=f_1=f_2$ approximately for oscillators numbered I, II, V and VI; for the others, f_0 differs from f_2 by ± 15 percent approximately. The data taken with the former are more reliable, since for these the adhesive is without effect. The value of the term in σ_1 (Eq. 13) is 0.002 for oscillator V and 0.0035 for oscillator VI, so that the agreement in the observed values of s_{11}' serves to verify the correctness of this term.

TABLE III. *Values of the elastic moduli obtained in the test experiment.*

T°K	$s_{11}\times 10^{13}$ cm²/dynes I	II	III	IV
273.1	22.23	22.19	22.31	22.29
85	18.72	18.68	18.77	18.80

T°K	$s_{11}'\times 10^{13}$ cm²/dynes V	VI	VII	VIII
273.1	28.22	28.24	28.23	28.48

In conclusion, the author gratefully acknowledges his indebtedness to Dr. Paul F. Kerr for his assistance in the selection of specimen material; to the Physics Department of Columbia University for the facilities generously placed at his disposal; and to Dr. S. L. Quimby, who followed the progress of the research with helpful counsel and encouragement.

[12] C. Chree, Quart. J. Math. **24**, 340 (1890).
[13] M. v. Laue, Zeits. f. Physik **34**, 347 (1925).

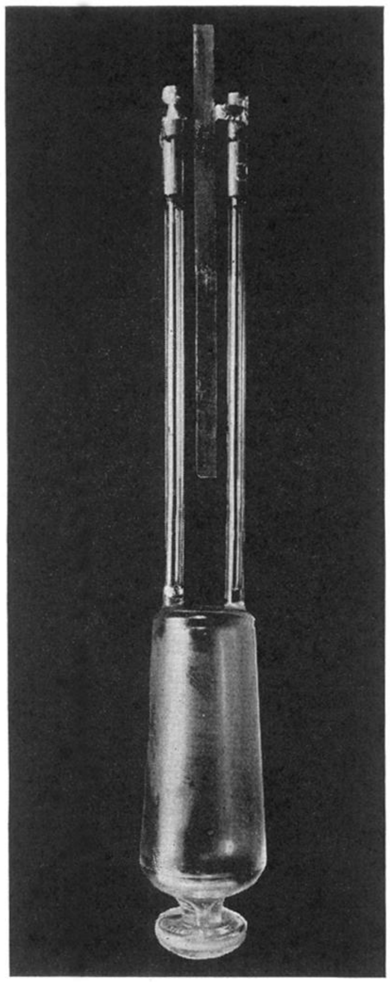

FIG. 3. A mounted oscillator.